WORK PLAN AND METHODOLOGY:

METHODOLOGY:

1) Spinel compounds can be prepared by many techniques. The compounds prepared by Co-precipitation method will be fine and small particle size and hence they can be use as a like better catalyst for some oxidation reaction. Therefore Co-precipitation method can be adopted in the present work. After its synthesis of spinels, they are characterized by XRD technique to find the formation of spinel phase. Then they are characterized by IR technique using FTIR instrument in the wavelength 400-2000 cm^{-1}.

Spinels show two strong bands in the IR characteristic of tetrahedral and octahedral cations. Then they are characterized by catalytic studies using H_2O_2 as a simple model reaction.

The present project makes use of the following A.R. Grade chemicals:

$ZnSO_{11}H_{14,}$ $CuSO_9H_{10,}$$CoSO_{11}H_{14}$,$FeSO_{11}H_{14}$,$Cr_2S_3O_{12,}$ ferric alum for the synthesis of spinels by co-precipitation method.

2. CO-PRECIPITATION METHOD:

In this method, the sulphates of appropriate metal ions will dissolved in water in required molar proportions.The hydroxides will be precipitated by adding sodium hydroxide and maintaining pH between 9 and 9.5. The precipitate shall be heated on water bath (90^oC) for 3 to 4 hours and oxidized by adding required amount of 30% H_2O_2 with constant stirring. The product will filtered washed and dried at 80^oC in a vacuum cryostat. The compound then heated at 1000^oC for 2 hours. The prepared spinel compound will characterised by using following techniques.

XRD Technique:

The product of solid state reactions will be usually in the form of powdered or sintered polycrystalline material. It the product is new phase, its X-ray powder pattern will not have been reported previously. X-ray analysis cannot give chemical analysis of the product. Other analytical techniques will be used to identify the chemical composition.

X-ray diffraction is an extremely important technique in the field of material characterization to obtain the information on atomic scale from both crystalline and non crystalline (amorphous) materials. The discovery of x- ray diffraction in 1912 (by max van laue) and its immediate application to structure determination in 1913 (by W.L. Bragg and his father W.H Bragg) paved the way for successful utilization of this technique to determine the crystal structures of the metals and its alloys, minerals, Inorganic compounds, and many more subsequently the technique of x-ray diffraction will also applied to drive information on fine structure of materials - crystalline size lattice strain, chemical composition, state of ordering etc.

X-ray crystallography is one of the most useful methods for exploring the nature of matter. X-ray diffractometer (XRD) is used to determine the phase content in many minerals and materials as a **r**esult it is used by researchers in wide range of disciplines. It is used as an adjunct to chemical analysis in the identification of the constituents of mixtures of crystalline phase. Each crystalline material has its own characteristic powder diffraction pattern, which serves as a finger print for its identification.

X-RAY AND THEIR PRODUCTION:

X-ray are high energy electromagnetic radiation, they have energy ranging from about 200 ev to 1 Mev, which outs between γ -ray and ultraviolet (UV) rays in electromagnetic spectrum . γ -ray and X-ray are identical, rays being more electromagnetic and shorter in wavelength than X-rays .But the main deference between them is way they are produced .X-ray produced by interactions between external beam of electrons in the shells of atom. on the other hand, x-rays are produced by changes in the nucleus of atom.

X-ray powder method serves to indicate, whether the reaction will completed, therefore by showing that the original reactants have disappeared and by showing that unwanted side products or intermediates have not formed.

The wave length of X-rays can returned as,

$$\lambda = (hc/E)$$

Where,

h=6.626x10^{-34} J-s (plank;s constsnt)

c=2.998x10^{8} m/s (speed of light)

E= energy of quantum of electromagnetic radiation

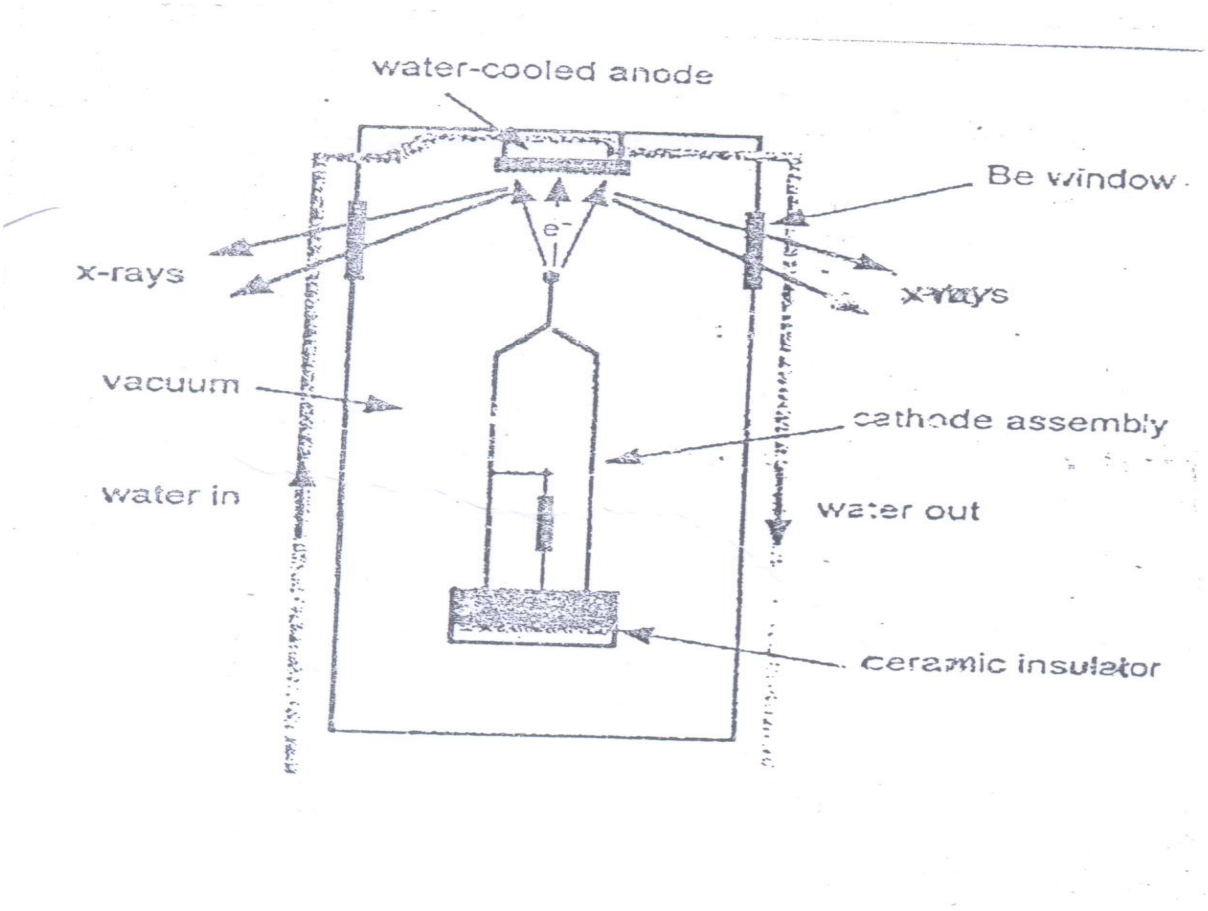

Fig. 2.1 schematic diagram of x-ray tube

X- rays are produced in the tube consisting of two metals electrodes enclosed in a vacuum chamber, as shown in fig. above, electrons are produced by heating a tungsten filament cathode. The cathode is at a very high negative potential, and the electrons are accelerated towards the anode, which is normally at ground potential. The loss of energy due to impact with metal anode is manifested as X-rays. Actually, a small percentage (less than 1%) of electron beam is converted to X-rays. the majority is dissipated as heat in the water cooled metal anode. Beryllium is used as a suitable material for windows as it is highly transparent to X-rays.

WORK PLAN :

In the first six months we will be studied about the spinel compounds and preparing preliminary starting compounds

In next two twelve months, will be synthesized & characterized the target spinel molecules by using different analytical techniques.

The last six months, study the application of synthesized spinel compounds and complying the data.